

सोडियम ट्राइपोलिफॉस्फेलेट,
निर्जल, तकनीकी — विशिष्टि

(दूसरा पुनरीक्षण)

**Sodium Tripolyphosphate,
Anhydrous, Technical —
Specification**

(Second Revision)

ICS 71.060

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FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Inorganic Chemicals Sectional Committee has been approved by the Chemical Division Council.

The standard was originally published in 1971 and was first revised in 1985 in the light of the progress made by the industry. In the first revision, changes were made in the requirement for sieve analysis and bulk density. The procedure for the gravimetric method for the determination of tripolyphosphate content was amended while the volumetric method was deleted. In this revision, alternate test methods for the determination of iron, copper and fluoride have been incorporated. In addition to this, alternate test methods for the determination of chlorides and phosphorus (as P_2O_5) have also been included. Further, a new requirement for lead (as Pb) along with its method of test has been incorporated in this standard.

Sodium phosphate, especially the polyphosphate, are chemicals of wide application. The newest member of the family of important sodium phosphates is sodium tripolyphosphate. The tripolyphosphate exhibits most of the useful properties of the other phosphates and none of their undesirable ones. It has a high sequestering action and is a good deflocculant like pyrophosphate. At present, the largest use of sodium tripolyphosphate is as a builder of synthetic detergents. Most of the built detergents sold today contain sodium tripoly and/or pyrophosphate. Among other important uses of sodium tripolyphosphate are ingredients of the formulation of boiler feed water treatment, purification of china clay, conditioning, of oil drilling mud, antipith agent in paper making, textile processing, dispersant in cements, etc.

Sodium tripolyphosphate is manufactured by neutralizing the phosphoric acid produced either by wet process or by thermal process. It is made from a mixture of one mole of monosodium orthophosphate and two moles of disodium orthophosphate, which is intimately mixed to get a good yield of the desired product. If the mixing is inadequate, combinations of pyro-, tripoly- and metaphosphates are formed. Currently the material in this country is being produced from phosphoric acid made by the wet process from imported rock phosphate. Keeping this in view the requirements have been laid down.

The composition of committee responsible for the formulation of this standard is given at Annex E.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (revised)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

SODIUM TRIPOLYPHOSPHATE, ANHYDROUS, TECHNICAL — SPECIFICATION

(Second Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for sodium tripolyphosphate, anhydrous, technical, for use as a detergent builder, and as a deflocculant.

2 REFERENCES

The Indian Standards given below contain provisions which through reference in this text, constitute provision of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards.

IS No.	Title
264 : 2005	Nitric acid — Specification <i>(third revision)</i>
265 : 1993	Hydrochloric acid — Specification <i>(fourth revision)</i>
324 : 1959	Ordinary denatured spirit
695 : 2020	Acetic acid — Specification <i>(fourth revision)</i>
1070 : 1992	Water for general laboratory use <i>(third revision)</i>
1997 : 2008	Laboratory glassware — Burettes <i>(third revision)</i>
3025 (Part 42) : 1992	Methods of sampling and test (physical and chemical) for water and wastewater: Part 42 Copper <i>(first revision)</i>
3025 (Part 60) : 2008	Methods of sampling and test (physical and chemical) for water and wastewater: Part 60 Fluoride <i>(first revision)</i>
4825 : 1982	Specification for liquid-In-Glass solid — Stem reference thermometers <i>(first revision)</i>

IS No.	Title
5470 : 2002	Dicalcium phosphate, animal feed grade — Specification <i>(first revision)</i>
16232 : 2014	Iron fortified iodized salt (double fortified salt) — Food grade — Specification

3 REQUIREMENTS

3.1 Description

The material shall consist essentially of sodium tripolyphosphate ($\text{Na}_5\text{P}_3\text{O}_{10}$). It shall be a white uniform free-flowing powder, free from visible impurities.

3.2 Sieve Analysis

When tested by the method prescribed in Annex A, the material shall conform to the following size grading:

Sieve Size	Percent by Mass
Retained on 500 micron IS Sieve	0.2, <i>Max</i>
Passing through 500 micron IS Sieve but	20, <i>Max</i>
retained on 150 micron IS Sieve	
Passing through 150 micron IS Sieve and	20, <i>Max</i>
retained on 75 micron IS Sieve	
Passing through 75 micron IS Sieve	60, <i>Min</i>

3.3 The material shall also comply with the requirements prescribed in Table 1 when tested according to the methods prescribed in Annex B. Reference to the relevant clauses of Annex B is given in col 4 of the Table.

3.4 Temperature Rise

The temperature rise, when determined by the method prescribed in Annex C, shall be as agreed to between the purchaser and the supplier.

Table 1 Requirements for Sodium Tripolyphosphate, Anhydrous, Technical
(Clause 3.3)

SI No.	Characteristic	Requirement	Method of Test (REF TO CL NO OF ANNEX B)
(1)	(2)	(3)	(4)
i)	Moisture, percent by mass, <i>Max</i>	1 . 0	B-2
ii)	Matter insoluble in water, percent by mass, <i>Max</i>	0..1	B-3
iii)	Phosphorus (as P_2O_5), percent by mass, <i>Min</i>	56.0	B-4
iv)	pH value	9.5 to 10.1	B-5
v)	Iron (as Fe_2O_3), percent by mass, <i>Max</i>	0.025	B-6
vi)	Copper (as Cu), ppm, <i>Max</i>	3	B-7
vii)	Lead (as Pb), percent by mass, <i>Max</i>	0.001	B-8
viii)	Tripolyphosphate content (as $Na_5P_3O_{10}$), percent by mass, <i>Min</i>	90.0	B-9
ix)	Chlorides (as NaCl), percent by mass, <i>Max</i>	1.25	B-10
x)	Fluorides (as NaF), parts per million, <i>Max</i>	600	B-11
xi)	Bulk density, g/ml	0.8 to 1.2	B-12

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in clean, dry and airtight steel drums or paper bags or as agreed to between the purchaser and the supplier. The containers shall not be opened until required for use and shall not remain open for a period longer than required for taking out the material.

4.2 Marking

4.2.1 Each container shall bear legibly and indelibly the following information:

- a) Name and grade of the material,
- b) Indication of the source of manufacture,
- c) Mass of the material in the container,

- d) Date of packing, and
- e) Batch number.

4.2.2 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act, 2016* and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

5 SAMPLING

The method of drawing representative samples of the material, the number of tests to be performed and the criteria for conformity of the material to the requirements of the specification shall be as prescribed in Annex D.

ANNEX A
(Clause 3.2)
SIEVE ANALYSIS

A-1 PROCEDURE

A-1.1 Clean and thoroughly dry the specified sieves. Nest the sieves in order with 500-micron IS Sieve on top. Weigh accurately 100 g of the well-mixed material and transfer it to the top sieve. Shake the sieves simultaneously and occasionally tap the bottom sieve on a rubber cushion. When the portion of the material passing through the bottom sieve appears to be negligible, remove the top sieve and shake the sieve separately over a clean glazed paper to ensure that no

more of the material passes through. Transfer from the paper any material that has passed through 500 micron IS Sieve to 150 micron IS Sieve using a camel hair brush. Repeat the procedure with 150 micron IS Sieve and 75-micron IS Sieve. Transfer the residue on the various sieves to convenient tared containers with the aid of a camel hair brush and weigh each separately.

A-1.2 Calculation

Calculate the masses on the basis of percent by mass of the material.

ANNEX B

(Clause 3.3)

METHODS OF TEST FOR SODIUM TRIPOLYPHOSPHATE, ANHYDROUS, TECHNICAL

B-1 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be used in tests.

NOTE — ‘Pure chemicals’ shall mean chemicals that do not contain impurities which affect the results of analysis.

B-2 DETERMINATION OF MOISTURE

B-2.1 Procedure

Weigh accurately about 5 g of the material in a clean wide-mouth glass weighing bottle, previously dried and weighed. Place the weighing bottle in an oven maintained at 150 ± 2 °C for two hours. Cool in a desiccator and weigh with the lid on. Heat again for 30 min, cool and weigh. Repeat this process until the loss in mass between the successive weighings is less than one milligram. Record the lowest mass obtained. Reserve the dried material for subsequent tests.

B-2.2 Calculation

Moisture, percent by mass =

$$\frac{100 (M_1 - M_2)}{M_1 - M}$$

where

M_1 = mass in g, of the weighing bottle with the sample before drying;

M_2 = mass in g, of the weighing bottle with the sample after drying; and

M = mass in g, of the empty weighing bottle.

B-3 DETERMINATION OF MATTER INSOLUBLE IN WATER

B-3.1 Procedure

Weigh 10g of the material accurately and dissolve it in 200 ml of water and boil for about 10 min. Filter the hot solution through previously dried and weighed sintered glass crucible G No. 3 or asbestos padded Gooch crucible. Wash the crucible with hot water till the filtrate is free from alkali. Place the crucible in an oven maintained at 110 ± 2 °C for two hours. Cool the crucible in a desiccator and weigh.

B-3.2 Calculation

Matter insoluble in water, percent by mass =

$$\frac{100 (M_2 - M_1)}{M}$$

where,

M_2 = mass in g, of the crucible after filtration;

M_1 = mass in g, of the crucible before filtration; and

M = mass in g, of the material taken for the test.

B-4 DETERMINATION OF PHOSPHORUS

B-4.1 Method A

B-4.1.1 Outline of Method

Total phosphorus is determined volumetrically by freshly hydrolyzing the material with dilute hydrochloric acid and titrating with sodium hydroxide solution.

B-4.1.2 Apparatus

B-4.1.2.1 Beaker — 250 ml capacity, marked at the 100 ml level.

B-4.1.2.2 pH meter

B-4.1.3 Reagents

B-4.1.3.1 Dilute hydrochloric acid — 5 N.

B-4.1.3.2 Buffer solution — pH 10.

B-4.1.3.3 Mixed indicator solution — Mix equal volumes of bromocresol green (0.1 percent in water or 20 percent alcohol), phenolphthalein (0.5 percent in 60 percent alcohol), and absolute alcohol.

B-4.1.3.4 Sodium hydroxide solution — 5 N approximately and 0.1 N freshly standardized.

B-4.1.4 Procedure

Weigh accurately about 3.5 g of the material into a beaker. Dissolve in water, transfer to a 500 ml volumetric flask, dilute to the mark and shake to mix.

Pipette out 75 ml into a 250 ml beaker marked at the 100 ml level. Add 15 ml of dilute hydrochloric acid and dilute to 100 ml. Introduce a boiling rod, cover with a watch-glass, and boil gently for 30 min. Add 0.5 ml of the mixed indicator, and neutralize the solution with sodium hydroxide solution (5 N) to a blue-green colour. Add dilute hydrochloric acid (0.5 N) until the indicator changes to yellow (about 0.5 ml is usually needed).

B-4.1.4.1 Dilute to 100 ml, if necessary, and boil for 15 min to expel carbon dioxide. Cool to room temperature. Wash and remove cover glass and wash boiling rod and walls of the beaker. Dilute to 100 ml. Place the beaker on the pH meter assembly. With thorough stirring (preferably mechanical) add standard sodium hydroxide solution (0.1 N) until the pH reaches 4.1. Alternatively, add the alkali in portions of about 0.2 ml, reading the burette after each addition, and interpolate to find the burette reading corresponding to pH 4.1.

B-4.1.4.2 Titrate to pH 8.8 with standard sodium hydroxide solution (0.1 N) interpolating as above, if desired. The final volume shall be about 150 ml; hence, excessive rinsing of the burette tip, electrodes, etc. should be avoided.

B-4.1.5 Calculation

Total phosphorus (as P_2O_5) =

$$\frac{T \times f \times 70.98 \times 500 \times 100}{10000} \frac{75}{M} \frac{T \times f \times 4.732}{M}$$

where,

T = difference in volume in, sodium hydroxide solution required in **B-4.1.4.1** and **B-4.1.4.2**;

f = factor of N/10 sodium hydroxide solution (see **B-4.1.6**); and

M = mass in g, of the material taken for the test.

B-4.1.6 Standardization of N/10 Sodium Hydroxide Solution — Analyze a sample of pure anhydrous sodium pyrophosphate ($Na_4P_2O_7$) by proceeding exactly as above except for accurately weighing 3.8 g of sample instead of 3.5 g.

$$\text{Total Phosphorus (as } P_2O_5) = \frac{T \times f \times 4.732}{M} = 53.38$$

$$\frac{53.38 \times M}{4.732 \times T}$$

where

T and f = same as in **B-4.1.5**, and

M = mass in g, of pure sodium pyrophosphate taken.

NOTE — If pure anhydrous sodium pyrophosphate is not available, pure potassium dihydrogen orthophosphate crystals may be used for standardization of N/10 sodium hydroxide solution, then f would be:

$$\frac{52.17 \times M}{4.732 \times T}$$

B-4.2 Method B (Spectrophotometric Method)**B-4.2.1 Outline of the Method**

A dilute solution of sodium tripolyphosphate is treated with an acidified molybdoavanadate reagent to form the yellow coloured molybdoavanado phosphoric acid complex.

$(NH_4)_5 PO_4 \cdot NH_4 VO_3 \cdot 16MoO_3$ with $NH_4 VO_3$, and $(NH_4)_6 Mo_7 O_{24} \cdot 4H_2O$

The reaction is sensitive to variations in acidity, and care must be taken to hold the acidity within limits. As the heat intensifies the colour, sample, and the standard must be at same temperature when the absorbance is measured.

B-4.2.2 Reagents

B-4.2.2.1 Solution A — Dissolve 1.12 g of ammonium metavanadate into water and then add 250 ml nitric acid.

B-4.2.2.2 Solution B — Dissolve 27 g of ammonium molybdate in water. If the solution is turbid add little NH_3 solution till the solution is clear. Mix solution B with A and dilute to 1 litre with water. Store the reagent in ambered coloured bottle.

B-4.2.2.3 Standard P_2O_5 solution — Dissolve 19.174 g of potassium dihydrogen phosphate (KH_2PO_4) in

1 litre volumetric flask with water. Add 2-3 ml of HNO_3 in order to preserve the solution. Dilute to the mark and mix well. One millilitre of this solution contains 10 mg of P_2O_5 .

B-4.2.2.4 Standard solutions of 4 mg and 5 mg of P_2O_5 per 10 ml of solution — Transfer 40 ml and 50 ml of original standard solutions by means of burette into two different 1-litre capacity volumetric flasks and dilute to the mark with water. The solutions are 10 ml = 4 mg and 10 ml = 5 mg of P_2O_5 , respectively.

B-4.2.3 Procedure

Transfer an aliquot of solution from the stock solution containing 40-50 mg of P_2O_5 into a 100 ml measuring flask. Dilute to the mark with water. From this flask transfer 10 ml of the solution into another 100 ml flask, dilute with water and add 20 ml of the reagent A, dilute to the mark with water, mix well and allow it to stand for half an hour.

Similarly transfer 10 ml aliquots of standard solutions containing 4.0 mg and 5.0 mg of P_2O_5 to 100 ml volumetric flasks, dilute with water, add 20 ml of reagent A, dilute to the mark with water and allow to stand for half an hour.

Adjust the spectrophotometer to read 70 percent transmittance (T) for the 4.0 mg standard using 10 mm cell at 420 nm wave length. Determine the percentage of T for the 5.0 mg standard. Similarly determine the percentage of T for the sample. Find out the log T from the table, plot a graph of absorbance against mg P_2O_5 and from the graph determine the mg P_2O_5 in the sample or calculate the mg of P_2O_5 in the sample by calculation.

B-4.2.4 Calculation

P_2O_5 , percent =

$$\frac{[\text{mg } \text{P}_2\text{O}_5 \times (\text{dilution factor})] \times 100}{\text{mass in mg of sample present in aliquot}}$$

Phosphoric acid (as H_3PO_4), percent by mass = percent $\text{P}_2\text{O}_5 \times 1.3803$

B-5 DETERMINATION OF pH

B-5.1 Apparatus

B-5.1.1 pH Meter — A suitable pH meter fitted with glass and calomel electrodes.

B-5.2 Procedure — Weigh 1.0 g of the material into a 150 ml beaker, add 100 ml of freshly boiled and cooled water and stir to dissolve. Measure the pH of the solution after 10 minutes with the help of a pH meter.

B-6 DETERMINATION OF IRON

B-6.1 Method A

B-6.1.1 Outline of Method — Iron is determined colorimetrically by measuring the transmittancy of reddish purple colour produced by ferrous and ferric ion with thioglycollic acid at pH 10.

B-6.1.2 Apparatus

B-6.1.2.1 Photoelectric absorptiometer

B-6.1.3 Reagents

B-6.1.3.1 Dilute hydrochloric acid — 5 N.

B-6.1.3.2 Sodium citrate solution 1 M — 29.4 g per 100 ml. Alternatively, ammonium citrate solution (2 M), 43.0 g per 100 ml, may be used.

B-6.1.3.3 Ammonium hydroxide — 10 N.

B-6.1.3.4 Thioglycollate reagent — Add 20 ml of ammonium hydroxide to 30 ml of water, then add a mixture of 10 ml of thioglycollic acid and 40 ml of water.

B-6.1.3.5 Standard iron solution — Dissolve 0.4911 g of ferrous ammonium sulphate $\text{FeSO}_4(\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$ in water, add 10 ml of dilute sulphuric acid (10 percent v/v). Dilute to exactly 100 ml in a volumetric flask. Take 25 ml of this solution into a 250 ml volumetric flask and dilute to the mark with water. One millilitre of this solution is equivalent to 0.1 mg of iron (as Fe_2O_3).

B-6.1.4 Procedure — Weigh accurately 1 g of the material into a 150 ml beaker. Add 20 ml of water and 5 ml of dilute hydrochloric acid. Introduce a boiling rod, cover with a watch-glass and boil gently for 15 to 20 min. Cool and transfer to a 100 ml volumetric flask and dilute to about 70 ml. Place 5 ml of dilute hydrochloric acid and 65 ml of water in a second flask as blank.

B-6.1.4.1 To each flask add 4 ml of sodium citrate solution (or 2 ml of ammonium citrate solution), 5 ml of thioglycollate reagent and 15 ml of ammonium hydroxide, swirling after each addition. Dilute to the mark and mix well.

B-6.1.4.2 Balance the absorptiometer at blank using green filter No. 4 and 4 cm cells. (Use 1-cm cells if the colour is too strong). Find the reading with the sample solution. Read off milligram iron from the standard calibration graph.

B-6.1.4.3 Standard calibration graph — Pipette known volumes of standard iron solution into each of 100 ml volumetric flasks containing 5 ml of dilute

hydrochloric acid. Proceed as in **B-6.1.4.1**. Suitable aliquots are 0, 1.0, 1.5, 2.0, 2.5 and 3.0 ml. Take the readings. Plot graph with milligram iron as ordinates and absorptiometer readings as abscissae. Draw a line through points and extend through the origin. Label the graph with particulars of filters and cells used.

B-6.1.5 Calculation

$$\text{Iron (as } \text{Fe}_2\text{O}_3\text{), percent by mass} = \frac{M_1}{M}$$

where

M_1 = mass in mg, of iron (Fe_2O_3) read from the graph; and

M = mass in g, of the material taken for the test.

B-6.2 Method B (Spectrophotometric Method)

B-6.2.1 Outline of the Method — Ferrous iron reacts with o-phenanthroline between pH 3 and 9 in the ratio of 1 : 3 to give a stable, orange-red coloured complex which has maximum absorbance at 510 nm. Silver and bismuth give precipitates. Copper, cadmium, mercury and zinc reduce the intensity of the colour. The effect of these ions can be overcome by using excess reagent.

B-6.2.2 Apparatus — Spectrophotometer.

B-6.2.3 Reagents

B-6.2.3.1 Standard iron solution — Dissolve 7.02 g of ferrous ammonium sulphate $\{\text{FeSO}_4(\text{NH}_4)_2\text{SO}_4\cdot 6\text{H}_2\text{O}\}$ in water containing 10 ml of concentrated sulphuric acid and dilute with water to 1 000 ml. One millilitre of this solution contains 1.0 mg of iron (as Fe). It may be diluted suitably to contain 10, 25 or 100 μg of iron per millilitre.

B-6.2.3.2 Hydroxylamine hydrochloride solution — Dissolve 10 g in water and dilute to 100 ml. This solution should be freshly prepared.

B-6.2.3.3 o-Phenanthroline solution — Dissolve 0.1 g of o-phenanthroline in 50 ml of ethyl alcohol and dilute to 100 ml with water. Mix well and store in a dark bottle with a ground-glass stopper.

B-6.2.3.4 Sodium acetate — acetic acid buffer solution — Dissolve 28 g of glacial acetic acid in water and dilute to 500 ml. Similarly dissolve 68 g of sodium acetate in 500 ml of water. At the time of use, prepare the required quantity of buffer solution by mixing equal volumes of the two solutions.

B-6.2.3.5 Dilute sulphuric acid — Approximately 4 N.

B-6.2.3.6 Dilute ammonium hydroxide

B-6.2.3.7 Preparation of standard graph — Take 5 ml of standard iron solution (10 $\mu\text{g}/\text{ml}$) into a 100 ml beaker. Add 10 ml of water, 1 ml of hydroxylamine

hydrochloride solution, 1 ml of dilute sulphuric acid and 5 ml of orthophenanthroline solution. Add sufficient ammonia solution to adjust the pH of the solution to 5 and then add 5 ml of buffer solution. Adjust the pH to 5 again (if necessary). Transfer the contents to a 100 ml volumetric flask and dilute to the mark with water. Keep aside the flask for 30 min to develop red colour fully. This solution corresponds to iron concentration of 0.05 mg/100 ml. Determine the optical density of this solution on the spectrophotometer at 510 nm. Similarly prepare solutions of iron concentrations 0.10 mg, 0.15 mg, 0.20 mg, 0.25 mg and 0.30 mg per 100 ml. Determine optical densities of these solutions and plot a graph of concentrations against corresponding optical densities.

B-6.2.4 Procedure

Weigh accurately 10 g of the material and transfer it into a one-litre conical flask. Add 150 ml of water and sufficient hydrochloric acid to dissolve the sample. When effervescence ceases, boil the contents to evolve out carbon dioxide. Cool to room temperature and dilute to 250 ml in a volumetric flask. Take 25 ml of the solution in a 100 ml beaker, add 1 ml of hydroxylamine hydrochloride solution and 5 ml of orthophenanthroline solution. Adjust the pH of the solution to 5 and then add 5 ml of buffer solution. Adjust the pH to 5 again (if necessary) and dilute to the mark with water. Keep the contents for 30 minutes to develop the red color fully and then determine the optical density on the spectrophotometer at 510 nm. From the standard graph, read the corresponding concentration of iron per 100 ml of the solution.

B-6.2.5 Calculation

$$\text{Iron content, percent by mass} = \frac{c \times d \times 100}{M}$$

where

c = concentration of iron in mg, per 100 ml of the sample solution;

d = dilution factor; and

M = mass in mg, of the sample taken for analysis.

NOTE — If the sample solution is strongly red coloured suitably less volumes, namely, 5 ml or 10 ml may be taken for analysis.

B-6.3 Alternatively, Iron can also be determined as per the method referred in Annex A of IS 16232 by use of spectrophotometer.

B-7 DETERMINATION OF COPPER

B-7.1 Method A

B-7.1.1 Outline of Method — Copper is determined colorimetrically by measurement of the colour produced by the material with zinc dibenzylidithiocarbamate. The value is read off from a standard calibration graph.

B-7.1.2 Apparatus

B-7.1.2.1 Separating funnels — 100 ml capacity. Rinse the funnel with dilute hydrochloric acid and with water before use. Allow to drain, then dry the stems with pure absorbent cotton wool and place a small plug of cotton wool in each stem.

B-7.1.2.2 Photoelectric absorptionzeter — With 1 cm cells.

B-7.1.3 Reagents

B-7.1.3.1 Ammonium hydroxide — 10 N approximately.

B-7.1.3.2 Dilute hydrochloric acid — 5 N.

B-7.1.3.3 Zinc Dibenzylthiocarboinate Solution — 0.04 percent solution in carbon tetrachloride.

B-7.1.3.4 Standard copper solution — Weigh 0.491 g of copper sulphate pentahydrate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) in a weighing bottle. Wash it into a 250 ml volumetric flask. Add 3 ml of 18 N sulphuric acid, dilute to about 100 ml, and swirl until the salt has completely dissolved. Dilute to the mark and mix well. This is the stock solution, one millilitre of which contains 0.5 mg of copper (Cu). Take 3 ml of 18 N sulphuric acid and about 200 ml of water in a 1000 ml volumetric flask. Carefully pipette 5 ml of the stock copper solution into the flask. Dilute to the mark and mix well. One millilitre of this solution contains 2.5 µg of copper (Cu).

B-7.1.4 Procedure

B-7.1.4.1 Weigh accurately 5 g of the material into a 250 ml beaker. Take a smaller mass if the copper is expected to exceed 3 parts per million. Add 30 ml of dilute hydrochloric acid and boil gently for 15 min. Place 30 ml of dilute hydrochloric acid in a second beaker as blank and continue with both. Allow to cool, then add ammonium hydroxide until the solution is alkaline to litmus paper. Add dilute hydrochloric acid dropwise until the solution is acidic, then add 3 ml in excess.

B-7.1.4.2 Transfer to a 100 ml separating funnel and, if necessary, dilute to 50 ml. Add 10 ml of zinc dibenzylthiocarboimate solution and shake vigorously for at least 2 min. Allow the layers to separate, then unstopper the funnel and run the lower layer through a plug of cotton wool into a 1 cm cell, using, the first few drops to rinse out the cell. Adjust the photometer at zero at 440 µm (blue photocell, plain glass filter) with a cell of zinc dibenzylthiocarboimate solution in the light path. Then measure the sample and the blank. Read off parts per million copper from the standard calibration graph.

B-7.1.4.3 Standard calibration graph — Take five 100 ml separating funnels, and wash thoroughly with hydrochloric acid and then with water. In each funnel place 3 ml of dilute hydrochloric acid (5 N). Using pipettes or a micro burette, measure respectively: 0, 2, 5, 10 and 15 ml of standard copper solution. Dilute to 50 ml and proceed as in **B-7.1.4.2**. Subtract a constant from each observed colorimeter reading so that the blank solution with no added copper corresponds to a reading of 0. Convert volumes of standard copper solution to micrograms of copper by multiplying by 2.5. Plot a graph with micrograms as ordinates and adjusted colorimeter readings as abscissae. Draw a line through the points and through the origin. Label the graph with particulars of cells and filters.

B-7.1.5 Calculation

$$\text{Copper (Cu), parts per million} = \frac{M_1}{M}$$

where

M_1 = mass in micrograms, of copper read off from the graph; and

M = mass in g, of the material taken for the test.

B-7.2 Method B (Spectrophotometric Method)**B-7.2.1 Principle**

Copper present in the sample is reduced with the help of ascorbic acid and by addition of 2,2'-biquinolyl a violet coloured complex is formed.

B-7.2.2 Reagents

Sodium sulphate, hydrochloric acid solution, amyl alcohol, (+) tartaric acid, sodium hydroxide, L-ascorbic acid (100 g/l), 2,2'-biquinolyl (0.5 g/l), bromine water-saturated solution, copper standard solution (0.1 g/l), indicator paper (narrow range) pH 5.5 to pH 7.0, Methyl orange indicator (0.5 g/l) aqueous solution.

B-7.2.3 Apparatus

Spectrophotometer, photoelectric absorptiometer with maximum transmission at a wavelength of 545 nm, Optical cells, 4 cm optical path.

B-7.2.4 Procedure**B-7.2.4.1 Test portion**

Weigh about 10g of the sample to the nearest 0.1 g.

B-7.2.4.2 Blank test

Carry a test with a blank as the determination follows the procedure.

B-7.2.4.3 Preparation of the calibration graph

Take six 500 ml separatory funnel and introduce the standard copper solutions.

Standard Copper Solutions

Standard Copper Solutions (ml)	Mass of Copper in μg
0	0
2	20
4	40
6	60
8	80
10	100

To all the funnels add 400 ml water and 2 ml of tartaric acid. pH of the solution is adjusted around 6.0 using sodium hydroxide. Add about 2 ml of ascorbic acid and mix the contents thoroughly. Allow the mixture to stand for 5 min. Add 2,2'-biquinolyl solution (10 ml) and mix well. Now try to extract the copper complex using two 20 ml of amyl alcohol portions. Transfer the extract to 100 ml beaker. Add sodium sulphate (2 g) to extract and remove water traces by stirring thoroughly.

In the next step, filter dry extract into 50 ml one mark volumetric flask. Wash residual sodium sulphate using 2 ml two portions of amyl alcohol. Dilute the mark using amyl alcohol by transferring to volumetric flask and mix well.

Spectrophotometric Measurements

Measurements are made using spectrophotometer or photoelectric absorptiometer at a wavelength of 545 nm. Measurements are made using filters and adjusting the instruments properly to zero absorbance using amyl alcohol as blank solution.

B-7.2.4.4 Determination of copper

Preparing Test solution

To a 400 ml beaker, transfer the test portion. Add 1 drop of methyl orange and 100 ml water. Using Hydrochloric acid, neutralize the solution adding 5 ml in excess. Add bromine water (10 ml) in excess and boil the solution till it is free from bromine. Allow the solution to cool. To a 500 ml separatory funnel fitted with stopper, transfer the contents of the beaker and add 1 ml of HCl solution to it. After the sample preparation and colour development, photometric measurements are made on the test samples using blank test solution (by adjusting the instrument to zero absorbance).

B-7.2.4.5 Calibration graph

The graph for the above standard solutions is plotted between absorbance vs concentration. Take the absorbance of all the prepared solutions and then deduce the concentration of copper in the unknown solution using a calibration graph.

B-7.2.5 Expression of Results

Determine the concentration of copper corresponding to the absorbance value. The copper content expressed as mg/kg is given by:

$$\frac{m_1 - m_2}{1000} \times \frac{1000}{m_o} = \frac{m_1 - m_2}{m_o}$$

where

m_o = is the mass of the test sample;

m_1 = is the mass of copper in test solution (in μg); and

m_2 = is copper in blank solution.

B-7.3 Alternatively, Copper can be determined as per the method referred in clause 5 of IS 3025 (Part 42) NEOCUPROINE METHOD.

B-8 DETERMINATION OF LEAD

B-8.1 Principle

A sample containing microgram quantities of lead is extracted with dithizone solution in chloroform. The extraction is carried out in the presence of strong ammoniacal citrate-cyanide reducing agent (pH 10 to 11.5). The quantity of lead present in the sample is determined spectrophotometrically by measuring the absorbance at 510 nm of the chloroform extract containing the lead dithizonate complex.

A-8.2 Minimum Detection Limit

1.0 μg Pb/10 ml dithizone solution (extract).

A-8.3 Apparatus

A-8.3.1 Spectrophotometer; for use at 510 nm with a path length of 1 cm or longer.

A-8.3.2 pH meter

A-8.3.3 Standard Volumetric Glasswares

A-8.3.4 TEF Beaker; 100 ml for Acid Digestion.

A-8.3.5 Separatory Funnel; 250 ml and 500 ml.

All glasswares are to be cleaned with 1 : 1 HNO_3 , and rinsed thoroughly with distilled water.

A-8.4 Reagents

A-8.4.1 Quality of Reagents

Only analytical or equivalent grade reagents, unless specified otherwise, are to be used. All reagents are to be prepared in lead-free distilled water.

A-8.4.2 Stock Lead Solution

Dissolve 0.1599 g lead nitrate $[(\text{Pb}(\text{NO}_3)_2$, minimum purity, 99.5 percent (w/w)] in about 200 ml of water.

Add 10 ml concentrated HNO_3 and dilute to 1000 ml with water, 1.0 ml of this solution will contain 100 μg of Pb.

A-8.4.3 Standard Lead Solution

Dilute 2.0 ml of stock lead solution to 100 ml with water, 1.0 ml of this solution will contain 2 μg of Pb.

A-8.4.3.1 Nitric acid — Concentrated (18N).

A-8.4.3.2 Nitric acid — Dilute — 20 percent, v/v.

A-8.4.3.3 Ammonium hydroxide — Concentrated (14 N).

A-8.4.3.4 Ammonium hydroxide — Dilute 10 percent, v/v and 1 percent, v/v.

A-8.4.3.5 Citrate-cyanide reducing solution — Dissolve 200 g anhydrous ammonium citrate $[(\text{NH}_4)_2 \text{HC}_6\text{H}_5\text{O}_7]$, 10 g anhydrous sodium sulphite (Na_2SO_3), 5 g hydroxylamine hydrochloride, 20 g potassium cyanide (KCN) in water and dilute to 500 ml, and mix with one litre of concentrated NH_4OH .

CAUTION — KCN is a poisonous solution. Handle with extreme care and do not pipette by mouth.

A-8.4.4 Stock Dithizone Solution

Dissolve 25 mg dithizone in about 50 ml chloroform (CHCl_3) taken in a 200 ml beaker and filter through Whatman No. 42 (or equivalent) filter paper. Collect the filtrate and two washings (10 ml each) in a 250 ml conical flask. Transfer the combined filtrate to a 500 ml separatory funnel. Add about 100 ml 1 percent (v/v) NH_4OH solution, shake moderately for about 1 min. Transfer the CHCl_3 layer to another 250 ml separatory funnel retaining the orange-red aqueous layer in the 500 ml separatory funnel. Repeat the extraction (of the CHCl_3 layer) with 100 ml of 1 percent (v/v) NH_4OH solution, transfer the CHCl_3 layer to another 250 ml separatory funnel and the aqueous layer to the original 500 ml separatory funnel containing the first extract. One more repetition, of extraction and transferring to the main aqueous layer is carried out. To the combined aqueous extract in the 500 ml separatory funnel add 1 : 1 HC1 in 2 ml portions, mixing after each addition, until dithizone precipitation is complete and the solution is no longer orange-red. Extract the precipitated dithizone with three 25 ml portions of CHCl_3 . Dilute the combined extract to 250 ml with CHCl_3 , 1 ml of this solution will contain 100 μg of dithizone.

A-8.4.5 Working Dithizone Solution

Dilute 100 ml stock dithizone solution to 250 ml in a standard volumetric flask with CHCl_3 , 1 ml of this solution will contain 40 μg of dithizone.

A-8.5 Procedure

A-8.5.1 Sample Digestion

Digest all samples for lead as per standard digestion procedure using $\text{HNO}_3\text{-H}_2\text{SO}_4$ and $\text{HNO}_3\text{-HClO}_4$.

To 100 ml acidified sample ($\text{pH}=2$) add 20 ml of dilute (20 percent, v/v) HNO_3 , filter if required through a filter paper (Whatman No. 41 or equivalent), and transfer it to a 250 ml separatory funnel. Add 60 ml ammoniacal citrate-cyanide solution, mix and cool to room temperature. Add 10 ml of dithizone working solution. Shake the stoppered funnel vigorously for about 30s, allow to stand (to get two separate layers). Discard 1-2 ml CHCl_3 layer and then fill the absorption cell. Measure the absorbance at 510 nm using working dithizone solution as reagent blank.

A-8.5.2 Calibration Curve

Plot a calibration curve using at least five standard lead solutions, after adding 50 ml ammoniacal citrate-cyanide solution to the individual lead standard solutions and extracting the same with 10 ml of dithizone working solution.

A-8.6 Calculation

$\text{mg Pb/litre} = \mu\text{g} \text{ (in 10 ml extract obtained from calibration curve)}/ \text{Volume of sample (ml)}$

B-9 DETERMINATION OF TRIPOLYPHOSPHATE CONTENT ($\text{Na}_5\text{P}_3\text{O}_{10}$)

B-9.1 Outline of Method — The method is based on the reaction that sodium tripolyphosphate forms a complex with trisdiaminoethane cobaltic chloride at pH 3.5 selectively in the presence of other phosphates.

B-9.2 Reagents

B-9.2.1 Wash Solution — Dissolve 20 g isopropanol in 100 ml of water.

B-9.2.2 p-Nitrophenol Indicator Solution — Dissolve 0.1 g of p-nitrophenol in 100 ml of water. **B-9.2.3 Dilute Hydrochloric Acid** — 2 N approximately.

B-9.2.4 Acetate Buffer Solution — pH 3.6. Dissolve 52 to 53 ml of glacial acetic acid (see IS 695) and 6.16 g of anhydrous sodium acetate in water and dilute to 500 ml.

B-9.2.5 Trisencobalt Reagent — Dissolve 10 g of trisdiaminoethane cobaltic chloride (as prepared in B-8.2), after drying at 110°C, in water and dilute to 250 ml. Add 1 drop of toluene to prevent mould growth.

B-9.2.6 Isopropyl Alcohol

B-9.2.7 Sodium Trypolyphosphate, Hexahydrate — Thrice crystallized as prepared in **B-8.1.3**.

B-9.3 Preparation of Trisdiaminoethane Cobaltic Chloride

B-9.3.1 Reagents

B-9.3.1.1 Diaminoethane hydrate (ethylene diamine)

B-9.3.1.2 Concentrated hydrochloric acid — see IS 265

B-9.3.1.3 Cobalt chloride ($\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$)

B-9.3.1.4 Methylated spirit — see IS 324

B-9.3.2 Procedure

Weigh 261 g of diaminoethane hydrate into a 1 000 ml beaker. Partly neutralize with 85 ml of concentrated hydrochloric acid in 535 ml of water. Pour the mixture with good agitation into a solution of 250 g of cobalt chloride in 750 ml of water contained in a conical flask. Pass a vigorous stream of air into the solution through a gas distribution tube for 8 h.

Transfer the solution to a 2 000 ml beaker and evaporate on a steam-bath under a stream of air until crystals form on the surface of the solution. Cool the solution and add 150 ml of concentrated hydrochloric acid with good mechanical stirring. Warm until the crystals re-dissolve. Salt out the trisdiaminoethane cobaltic chloride by slowly adding, over a period of 1 hour, 300 ml of methylated spirit to the mechanically stirred solution. Cool and filter through a Buchner funnel with suction. Discontinue suction and wash by slurring with 150 ml of methylated spirit. Re-apply suction and filter. Repeat the washings three more times. Suck dry; spread out the crystals in an evaporating dish and allow the methylated spirit to evaporate.

Dissolve the crystals obtained in (b) above in 200 ml of boiling water in a 1 000 ml beaker on a hot-plate using mechanical stirring. If the crystals do not dissolve in this volume of water, add 20 ml portion of water, reheating to boiling after each addition, until complete solution is obtained. Remove from the source of heat and salt out with 300 ml of the methylated spirit as above. Filter and wash as before. If four washings do not give a colourless filtrate, continue washing until the filtrate is colourless.

Spread out the crystals in a thin layer in large evaporating dishes and allow to air-dry overnight. Then dry in an oven at 110 °C overnight.

NOTE — These crystals are hygroscopic and should be freshly dried each time they are weighed to make a new solution. Any yield of about 300 g of the dried crystals is obtainable.

B-9.4 Preparation of Pentasodium Triopolyphosphate, Hexahydrate ($\text{Na}_5\text{P}_3\text{O}_{10} \cdot 6\text{H}_2\text{O}$)

B-9.4.1 Procedure

Weigh 150 g of commercial sodium tripolyphosphate, using material of low metaphosphate content. Dissolve

in 1 200 ml of water, and filter to remove any suspended material. Add 300 ml of methylated spirit slowly, with mechanical stirring, over a period of 2 h. Separate the crystals of hexahydrate by filtration using a Buchner funnel fitted with a glass disc of medium porosity. Wash the crystals successively with 100 ml each of 25 percent, 50 percent, 75 percent and 90 percent (v/v) solution of methylated spirit in water.

Dissolve the hexahydrate in 900 ml of water and salt out with 225 ml of methylated spirit using the same procedure as above, again, filtering and washing the crystals with the same volumes of dilute methylated spirit. Repeat the recrystallization using 675 ml of water and 170 ml of methylated spirit. Recrystallize once more using 500 ml of water and 150 ml of methylated spirit. After filtering and washing, air-dry the crystals of hexahydrate for several days.

NOTE — The yield is about 60 g of $\text{Na}_5\text{P}_3\text{O}_{10} \cdot 6\text{H}_2\text{O}$. Store the hexahydrate in a tightly stoppered bottle. The purification procedure should not be interrupted for any length of time unless the material is in a crystalline form, to avoid hydrolysis. Do not allow it to remain in solution overnight.

B-9.5 Procedure for Determination of Tripolyphosphate Content

B-9.5.1 Transfer 3.500 ± 0.003 g of the material to a beaker. Dissolve in water, transfer to a 500 ml graduated flask, dilute to the mark and mix well. Pipette 50 ml of this solution into a 250 ml conical flask. Add 4 drops of *p*-nitrophenol indicator, and add dilute hydrochloric acid drop-wise until the solution is just acidic (that is, when the indicator becomes colourless). Add 10 ml of acetate buffer solution and 30 ml of isopropanol and then heat the solution to 40 °C.

B-9.5.2 Transfer 10 ml of trisencobalt reagent into a clean 10 ml burette. Add four 1 ml portion of the reagent from the burette and shake for 60 s between each addition, shake the flask for 15 min and then add the rest of the reagent in 1 ml portion, shaking for 60 seconds between each addition.

B-9.5.3 Reheat at 40 °C for 30 min. Filter through a medium porosity sintered glass crucible (which has previously been washed, dried and weighed).

B-9.5.4 Wash the precipitate with 2 percent isopropanol, wash solution to make it free from chloride (normally 6 to 8 times is required). Dry in an air oven at 98 °C-102 °C for 1 to 2 hours, cool in a desiccator and weigh till constant mass.

B-9.6 Calculation

Sodium tripolyphosphate content percent by mass (as $\text{Na}_5\text{P}_3\text{O}_{10}$) = $198.4 \times M$

where

M = mass in g, of the precipitate obtained.

NOTE — When the analysis obtained indicates less than 85 percent of sodium tripolyphosphate, take a 25-ml aliquot and add 0.2264 g of pure tripolyphosphate hexahydrate crystals, washed with 25-ml of water. Multiply the result by two and deduct 100 percent. If appreciable amounts of phosphate glass are present and indeed in any case where precipitation does not occur when the first 4 ml of reagent are added, this modification should always be used.

B-10 DETERMINATION OF CHLORIDES

B-10.1 Method A

B-10.1.1 Outline of Method — Chlorides are determined gravimetrically by precipitating with silver nitrate in nitric acid medium.

B-10.1.2 Reagents

B-10.1.2.1 Concentrated nitric acid — see IS 264.

B-10.1.2.2 Silver nitrate solution — 0.1 N approx.

B-10.1.2.3 Dilute nitric acid — 0.02 N approx.

B-10.1.3 Procedure

Weigh about 5 g of the material and wash into a 400 ml beaker. Dilute to 150 ml with water, and add 3 ml of concentrated nitric acid. Warm and stir until the sample dissolves. Cover the vessel with thick brown paper, to minimize exposing the subsequent precipitate to light. Add from a pipette, with constant stirring, 50 ml of silver nitrate solution. Heat the solution to nearly boiling, while stirring constantly and maintain it at this temperature until the precipitate coagulates and the supernatant liquor is clear (2 to 3 min). Make certain that the precipitation is complete by adding a few drops of silver nitrate solution to the supernatant liquor. If no further precipitate appears, place the beaker in the dark and allow to stand for 1 h. Filter the liquor through a tared sintered glass crucible G No. 4. Wash the precipitate three times by decantation with cold dilute nitric acid before transferring the precipitate to the crucible. Transfer the last particles with the aid of a rubber-tipped rod. Wash the precipitate at least ten times with small amounts of dilute nitric acid. Heat the crucible in an oven at 130 °C to 150 °C for 30 min. Cool in a dessicator and weigh. Repeat the operation till constant mass is obtained.

B-10.1.4 Calculation

Chlorides (as NaCl), percent by mass = $\frac{M_1}{M} \times 40.8$
where

M_1 = mass in g, of the precipitate obtained; and

M = mass in g, of the material taken for the test.

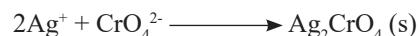
B-10.2 Method B

B-10.2.1 Outline

This method uses chromate ions as indicator in chloride ions estimation. These chloride ions are precipitated

as silver chloride. Any excess of titrant leads to the formation of a silver chromate precipitate.

The reactions are:



B-10.2.2 Apparatus — Conical flask, burette, pipette, measuring cylinder, dessicator and graduated cylinder.

B-10.2.3 Reagents — Standard silver nitrate, titrant, potassium chromate indicator.

B-10.2.4 Method

B-10.2.4.1 Preparation of 5 percent K_2CrO_4 (indicator):

Dissolve 1.0 g of K_2CrO_4 in 20 ml of distilled water.

Silver nitrate solution preparation: Transfer 9.0 g of silver nitrate in 500 ml volumetric flask and make up the volume with the help of distilled water. Concentration of the resultant solution is approximately 0.1 M. Standardization of silver nitrate solution can be done against NaCl.

B-10.2.4.2 Determination of Chloride in a sample

Sample was dried at 110 °C for about 1 h and it was then cooled in a dessicator. Sample was then weighed in 250 ml flasks and dissolved in 100 ml distilled water. To this, add small amount of sodium bicarbonate till the effervescence is ceased. Add around 2 ml of potassium chromate and the solution was titrated against silver nitrate till first appearance of red silver chromate is observed.

B-10.2.4.3 Standardization of silver nitrate

N_1V_1 (silver nitrate) = N_2V_2 (sodium chloride)

N_1 (silver nitrate) = N_2V_2 (sodium chloride) / V_1

B-10.2.4.4 Estimation of chloride sample

$\text{N}_1\text{V}_1 = \text{N}_3\text{V}_3$

$\text{N}_3 = \text{N}_1\text{V}_1 / \text{V}_3$

Strength = $\text{N}_3 \times \text{eq. wt, g/l}$

B-11 DETERMINATION OF FLUORINE BY DISTILLATION METHOD

B-11.1 Outline of the Method

Fluorine is separated as hydrofluosilicic acid by distillation with perchloric acid and determined by titration against standard thorium nitrate solution using sodium alizarinesulphonate as indicator.

B-11.2 Apparatus

B-11.2.1 The distillation apparatus (see Fig. 1) consists of a 250 ml distillation flask (A), side arm of which is connected to a condenser. The neck of the flask is fitted with a two-holder rubber stopper flask to a steam

generator (E), which is equipped with two discharges, one tube (F), to release steam and the other (D) to deliver steam into the distillation flask (A). The distillate is collected in a PVC beaker (500 ml).

B-11.2.2 Nessler Cylinders — 100 ml capacity with marks 100 ml and 50 ml

B-11.2.3 Thermometer — 0-150 °C.

B-11.2.4 Burette with Top — 10 ml capacity, graduation interval 0.05 ml (see IS 1997)

B-11.3 Reagents

B-11.3.1 Perchloric Acid — 70 percent.

B-11.3.2 Sodium Alizarine Sulphonate Indicator — Dissolve 0.02 g of sodium alizarinesulphonate in 100 ml of water and store in bottle.

B-11.3.3 Sodium Hydroxide Solution — Dissolve 10 g of sodium hydroxide pellets in water, cool and dilute to one litre. Mix well.

B-11.3.4 Dilute Hydrochloric Acid — Dilute 19.6 ml of concentrated hydrochloric acid to 1 000 ml with water. Mix well.

B-11.3.5 Standard Thorium Nitrate Solution — 0.02 N. Dissolve 2.940 9 g of thorium nitrate, $\text{Th}(\text{NO}_3)_4 \cdot 6\text{H}_2\text{O}$ in water and make up to one litre. Mix well.

B-11.3.6 Standard Sodium Fluoride Solution (1 ml = 0.1 mg NaF) — Dissolve 1.0 g of sodium fluoride (AR) previously dried at 120 °C to constant mass in one litre of water, in a volumetric flask, and further dilute 10 ml of this solution to 100 ml containing 20 ml of 1 N NaOH. This is the standard solution (1 ml = 0.1 mg NaF) which can be used for titration. Keep the solution in polyethylene bottle.

B-11.4 Procedure

B-11.4.1 Transfer 10.0 g of an accurately weighed sample to a 250 ml distillation flask, containing a few glass beads. Add 10 ml of perchloric acid.

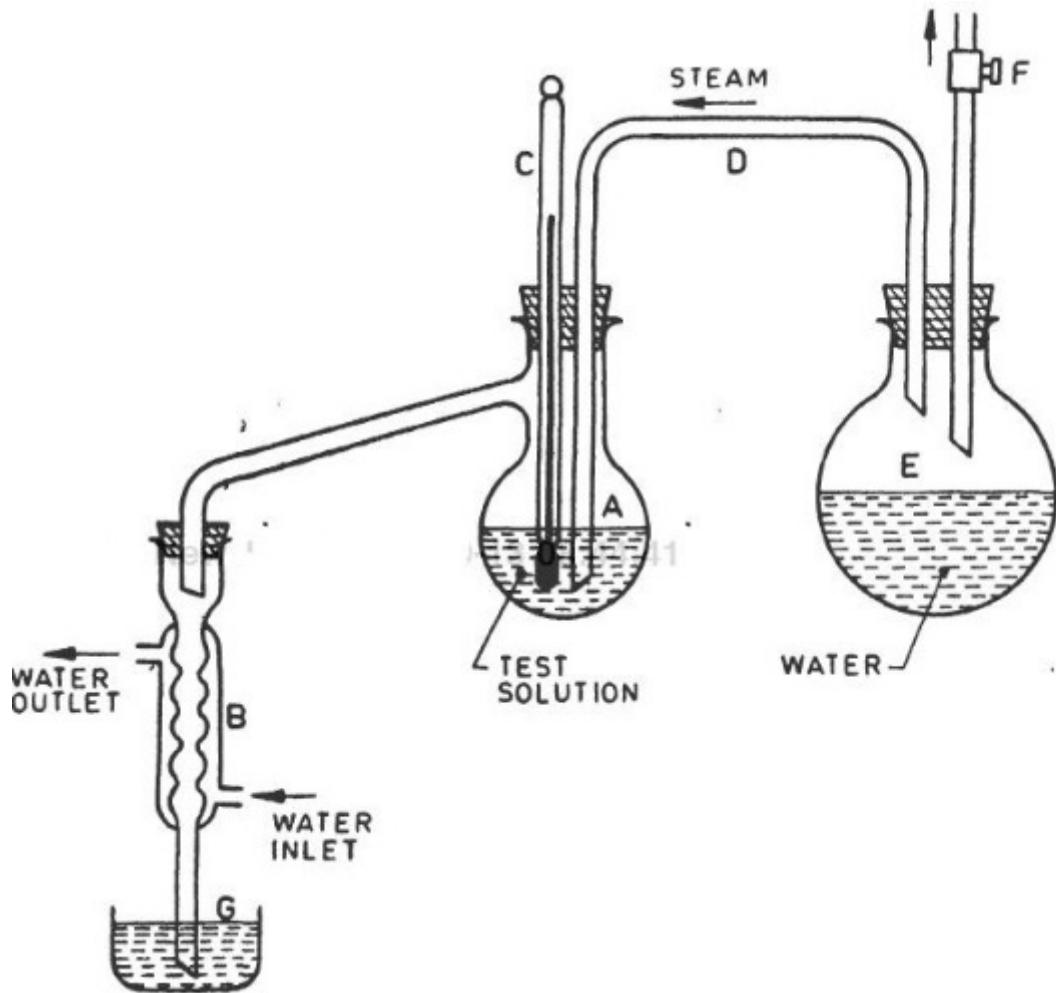


FIG. 1 APPARATUS FOR STEAM DISTILLATION FOR FLUORIDE DETERMINATION

B-11.4.2 Place a 500 ml PVC beaker under the condenser. Connect the apparatus as shown in Fig. 1, distillation flask (A) to the condenser and distil the solution until the boiling point reaches 140 °C. While this is being done, heat the water in the steam generator (E) to boiling but do not allow the steam to get in the flask (A). When the temperature reaches 140 °C admit steam into the flask (A). Maintain the temperature 140 °C ± 2 °C by adjusting the steam flow and heating of the flask (A). Distil at such a rate that no fumes or mist escape from the PVC beaker. Continue distillation until nearly 300 ml are collected in a beaker.

B-11.4.3 Transfer to a 500 ml volumetric flask. Make the volume upto the mark and mix well. Pipette out 50 ml aliquot into a 150 ml beaker.

Take 10 ml of standard sodium fluoride (1ml = 0.1 mg) solution in another 150 ml. Add 1 ml of sodium alizarinesulphonate indicator in each beaker. Adjust the pH of both the solution on pH meter to 3.1 using sodium hydroxide or dilute hydrochloric acid as the case may be. Transfer both the solutions into two different Nessler cylinders. Add thorium nitrate (0.020 N) solution from burette in standard fluoride solution until light pink colour appears without the formation of precipitate (suitable for comparison). Note the reading. Add thorium nitrate in sample solution till it matches in colour developed with standard fluoride solution. Again note the reading.

B-11.5 Calculation

$$\text{Sodium fluoride, } \frac{T \times F}{1000} \times \frac{500}{50} \times \frac{1000000}{M}$$

parts per million

$$= \frac{V \times F \times 10000}{M}$$

where

V = volume in ml, of thorium nitrate required for sample solution,

F = $\frac{\text{mg of NaF taken for comparison}}{\text{ml of thorium nitrate added to it}}$

M = mass in g, of sample taken for distillation.

B-11.6 Alternatively, fluoride can also be determined as per the method referred in Annex B of IS 5470 : 2002 by Ion Selective Electrode Method OR as per clause 6

of IS 3025 (Part 60) : 2008 by electrochemical probe method in which fluoride ion selective electrode is used.

B-12 BULK DENSITY

B-12.1 Apparatus

B-12.1.1 The apparatus is shown in Fig. 2. The base of the measuring cylinder A shall be ground flat and the empty measuring cylinder A together with the rubber bung shall weigh 250 ± 5 g. It shall be accurately calibrated to 250 ml with an error of less than one millilitre. The distance between zero and 250 ml graduation on the measuring cylinder A shall be not less than 220 mm and not more than 240 mm. The distance between the flat-ground part of the base of the measuring cylinder A and the rubber base pad B, when the measuring cylinder A is raised to the full height shall be 25 ± 2 mm.

B-12.1.2 Balance — Pans of the balance shall be at least 10 cm in diameter and the balance shall be sensitive to less than 0.1 g.

B-12.1.3 Rubber Base Pad — B shall have a shore hardness of 35 to 50.

B-12.2 Procedure — Weigh 100 g of the material in a paper and form it into a chute. Introduce the powder into the cylinder, which should be held at 45° to the vertical. Slip the powder gently and smoothly into the measuring cylinder without knocking or squeezing. Assemble the apparatus. With the thumb and four fingers of the hand, gently grasp the upper part of the cylinder and within one second lift it as far as 25 mm. Do not pick by knocking it against the upper stop. At the start of the next second, release the cylinder smoothly. Continue lifting and dropping until 50 complete drops have been given. Once in every 2 s a gentle twist of 10° should be given to the cylinder. Read off the volume.

B-12.3 Calculation

Bulk density (g/ml) = M/V

where

M = mass in g, of the material taken for the test; and

V = final volume in ml, of the material.

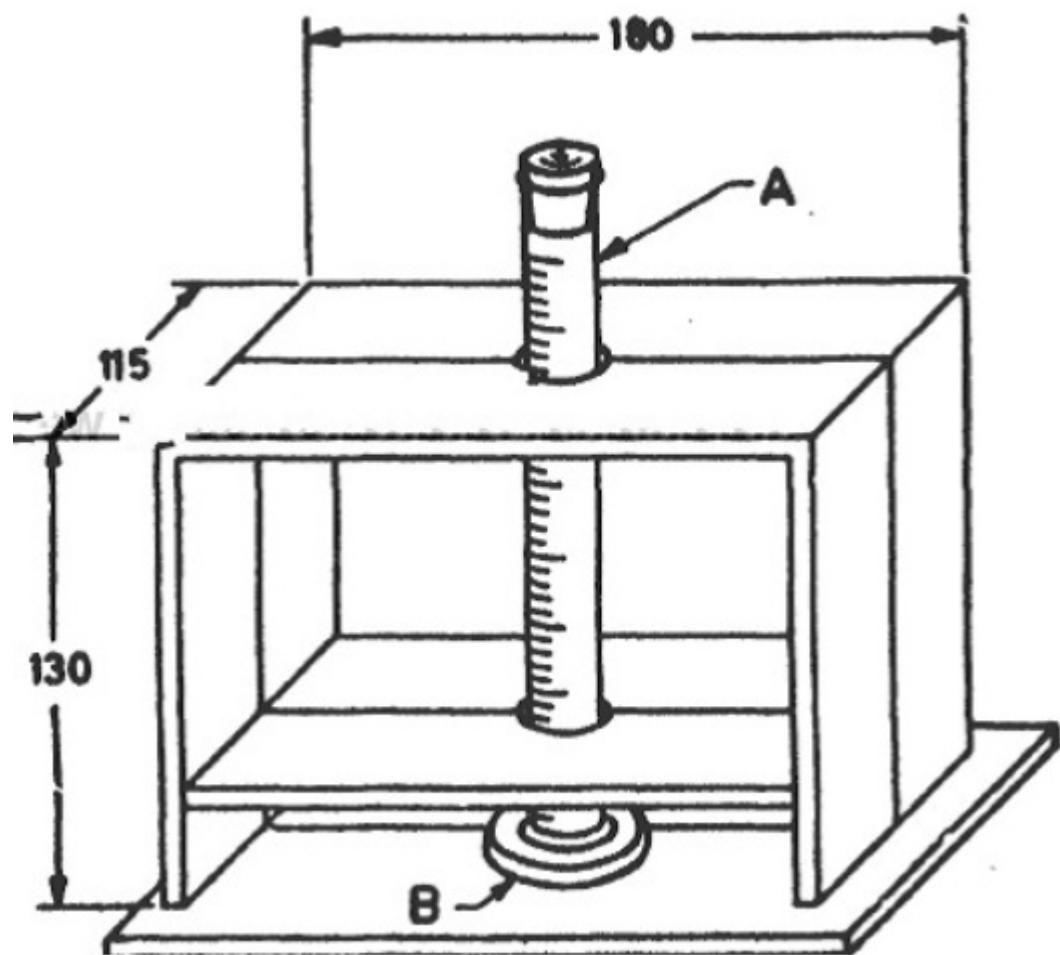


FIG. 2 APPARATUS FOR DETERMINATION OF BULK DENSITY

ANNEX C
 (*Clause 3.4*)
DETERMINATION OF TEMPERATURE RISE

C-1 GENERAL

There are two types of sodium tripolyphosphate, namely, phase I and phase II, both of which are found in commercial samples. The ratio of Phase I to Phase II determined by an empirical test based on the fact that Phase I hydrates more rapidly than Phase II. This test, called the temperature rise test has been calibrated by X-ray analysis.

C-2 APPARATUS

C-2.1 Thermos Flask — Wide-mouth vacuum flask, capacity about 1 200 ml; inner dia about 8 cm; height about 22 cm.

C-2.2 Beaker — Tall form 200 ml; height 10 cm; dia 6.5 cm.

C-2.3 Stirrer — A stirrer having two 3.5 cm diameter rings attached 2.5 cm apart to a handle and made from 0.3 cm stainless steel rod.

C-2.4 Thermometers — Two thermometers graduated from 0 to 50 °C, least count 0.1 °C and total length 550 mm (*see* schedule mark 22 of IS 4825).

C-2.5 Stop-watch

C-2.6 Measuring Cylinder — 25 ml capacity.

C-3 REAGENTS

C-3.1 Glycerine — Purity 96.5 ± 0.5 percent; specific gravity at 20°/20 °C, 1.2546 ± 0.0015.

C-4 PROCEDURE

C-4.1 Weigh 50.0 ± 0.1 g of glycerine into the dry beaker at room temperature and put 50.0 ± 0.1 g powdered sample at room temperature on top of the glycerine. Suspend the beaker in the neck of vacuum flask and insert the dry stirrer to the bottom of the beaker (*see* Fig. 3).

C-4.2 Stir with vertical strokes for 30 s at the slow speed (*see* Note under **C-4.4**) (to prevent dusting and loss of sample and to mix powder and glycerine properly). Then increase the stirring rate quickly to fast speed (*see* Note under **C-4.4**) and stir for $2\frac{1}{2}$ min.

C-4.3 Stop stirring. Clamp the dry thermometer in a central position with the tip 10 mm above the bottom of the beaker (lower it until it touches the bottom and lift 10 mm). Read and record the highest temperature of the paste that is constant for 30 s (T_1).

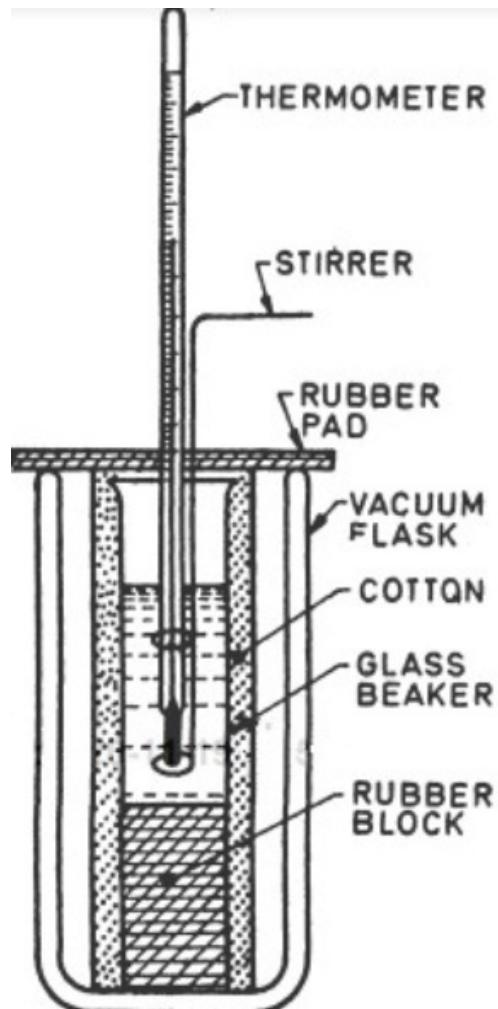


FIG. 3 APPARATUS FOR DETERMINATION OF TEMPERATURE RISE

C-4.4 Quickly add 25 ml of water at temperature (T_1) within ± 0.1 °C and then stir at slow speed for 5 seconds and then at fast speed for 1 min. Push the stirrer down to the bottom of the beaker. Observe the temperature without stirring until it has reached a maximum and decreased again at least 0.1 °C (T_2).

NOTE — Slow speed : about 90 complete cycles/minute.
 Fast speed : about 240 complete cycles/minute.

A complete cycle always means lifting up the bottom ring of the stirrer from the bottom of the beaker just to the surface of the mixture and pushing it back down until it touches the bottom again. Scrapping of the sides is also desirable.

C-5 CALCULATIONS**C-5.1** Temperature rise (deg C) = $T_2 - T_1$

where

 T_2 = maximum temperature deg C, and T_1 = paste temperature deg C.**C-5.2** Phase 1, percent by mass = (Temperature rise in deg C).**ANNEX D***(Clause 5.1)***SAMPLING OF SODIUM TRIPOLYPHOSPHATE, ANHYDROUS, TECHNICAL****D-1 GENERAL REQUIREMENTS OF SAMPLING****D-1.1** In drawing, preparing, storing and handling the samples, the following precautions shall be observed.**D-1.2** Samples shall not be taken at a place exposed to the adverse effects of weather.**D-1.3** The sampling instruments and sample containers shall be clean and dry,**D-1.4** Before drawing the samples, the contents of the selected containers shall be thoroughly mixed.**D-1.5** After filling, the sample containers shall be sealed and marked with relevant particulars.**D-2 SCALE OF SAMPLING****D-2.1** All the containers in a consignment of the material drawn from a single batch of manufacture shall constitute a lot. Each lot shall be tested separately for all the requirements of the specification.**D-2.2** The number of containers to be sampled at random from a lot depends on the size of the lot and shall be as given in Table 2.**Table 2 Number of Containers to be Selected For Sampling***(Clause 2.2)*

Lot Size (1)	No. of Containers to be Selected in the Sample (2)
Up to 50	3
51 to 100	4
101 to 150	5
151 and above	7

D-3 PREPARATION OF TEST SAMPLES**D-3.1** Draw with an appropriate sampling implement about 100 g of the material from different parts of each of the selected containers. These quantities are called individual samples representing the selected containers. These individual samples shall be kept in different sample containers which shall be marked with all details of sampling.**D-3.2** Equal quantities from each of the individual samples shall be taken and mixed together to form a composite sample weighing approximately 200 g.**D-4 NUMBER OF TESTS****D-4.1** Tests for the determination of phosphorus content and matter insoluble in water shall be conducted on each of the individual samples.**D-4.2** Tests for the remaining characteristics shall be conducted on the composite sample.**D-5 CRITERIA FOR CONFORMITY****D-5.1 For Individual Samples** — From the test results Mean (X) and Range (R) shall be calculated.

$$\text{Mean } (X) = \frac{\text{Sum of the test results}}{\text{Number of tests}}$$

Range (R) = Difference between the maximum and minimum value of the test results.**D-5.1.1** For phosphorus content the lot is deemed to conform to the specification if $X - 0.6 R$ is not less than the minimum value given in Table I and for matter insoluble in water the lot is deemed to conform to the specification if $X + 0.6 R$ is not greater than the maximum value given in Table 1.**D-5.2** For the remaining characteristics, all the test results shall satisfy the corresponding requirements given in col 3 of Table 1.

ANNEX E

(Foreword)

COMMITTEE COMPOSITION

Inorganic Chemicals Sectional Committee, CHD 01

<i>Organization</i>	<i>Representative(s)</i>
Central Salt and Marine Chemicals Research Institute, Bhavnagar	DR KANNAN SRINIVASAN (Chairman)
Alkali Mfrs Association of India, Delhi	SHRI K. SRINIVASAN SHRI SUBHASH TANDON (<i>Alternate</i>)
Bhabha Atomic Research Centre, Mumbai	DR A. V. R. REDDY DR S. N. ACHARY (<i>Alternate</i>)
Central Drugs Standard Control Organization DGQA, New Delhi	DR RAMAN MOHAN SINGH
Geological Survey of India, Kolkata	DR GURBACHAN SINGH SHRI B. S. TOMAR (<i>Alternate</i>)
Grasim Industries Ltd, Nagda	DR D. K. DAS DR SUBHAS CHANDRA (<i>Alternate</i>)
Gujarat Alkalies and Chemicals Ltd, Vadodara	SHRI ALOK SINGH SHRI PANKAJ GUPTA (<i>Alternate</i>)
Hindustan Lever Ltd, Mumbai	DR SUNIL SINHA
In personal capacity	VRINDARAJWADE SHRIMATI POORNAKALA (<i>Alternate I</i>) SATYAMOORTHY (<i>Alternate II</i>)
In personal capacity	DR A. N. BHAT
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Indian Institute of Chemical Technology, Hyderabad	SHRI ROHIT KUMAR MADHAVJI SHRI SATYAN ROHIT KUMAR (<i>Alternate</i>)
Ministry of Defence (DGQA), Kanpur	DR PRAVEEN R. LIKHAR DR RAJENDER REDDY (<i>Alternate</i>)
Ministry of Chemicals & Fertilizers	SHRI R. N. APARAJIT
National Chemical Laboratory, Pune	DR ROHIT MISRA
National Metallurgical Laboratory, Jamshedpur	DR DARBHA SRINIVAS DR PARESH DHEPE (<i>Alternate</i>)
National Mineral Development Corporation Ltd, Hyderabad	DR TRILOCHAN MISHRA SHRI DEVBRATA MISHRA (<i>Alternate</i>)
National Physical Laboratory, New Delhi	SHRI RAJAN KUMAR DR PRASHANT SHARMA
National Test House (NR)	DR NAHAR SINGH DR S. P. SINGH (<i>Alternate</i>)
Nirma Chemicals, Ahmedabad	DR Y. C. NIJHAWAN DR (Ms) A. BISWAS (<i>Alternate</i>)
Office of the Development Commissioner (MSME), New Delhi	SHRI R. A. JOSHI, DR K. C. PATHAK (<i>Alternate</i>)
Reliance Industries Ltd, Navi Mumbai	SHRIMATI SHIMLA MEENA SHRI SANTOSH KUMAR (<i>Alternate</i>) SHRI DHANAVADAN MODI

<i>Organization</i>	<i>Representative(s)</i>
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Central Drugs Standard Control Organization	DR RAMAN MOHAN SINGH
Indian Chemical Council (ICC)	DR U. SHETKAR
Delhi Jal Board	SHRI ASHUTOSH KAUSHIK
In Personal Capacity	SHRI D. K. JAIN
In Personal Capacity	DR AMITAVA DAS
Vaibhav Analytical Services, Ahmedabad	SHRI GAURANG OZA
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Member Secretary

SAGAR SINGH
SCIENTIST 'C' (CHD), BIS

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